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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : C04B 38/00, B01F 3/04, 5/04 | A1

(11) International Publication Number: WO 98/42637

(43) International Publication Date: 1 October 1998 (01.10.98)

(21) International Application Number: PCT/AU98/00196

(22) International Filing Date: 25 March 1998 (25.03.98)

(30) Priority Data:
PO 5808 25 March 1997 (25.03.97) AU

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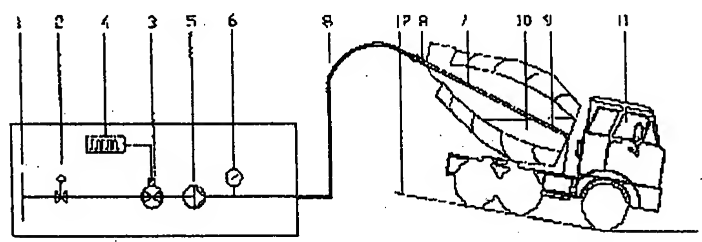
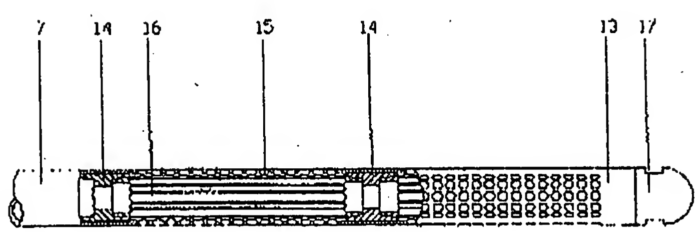
(81) Designated States: AL, AU, BA, BB, BG, BR, CA, CH, CN, CU, CZ, EE, GE, GW, HU, ID, IL, IS, JP, KP, KR, LC, LK, LR, LT, LV, MG, MK, MN, MW, MX, NO, NZ, PL, RO, SG, SI, SK, SL, TR, TT, UA, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: AERATED, LIGHTWEIGHT BUILDING PRODUCTS





(57) Abstract

This invention relates to Masonry Building Products inflated by an inert gas in such manner that the final Specific Gravity of the Product is lower than the original composition of components contained in the product. To inflate the aqueous slurry mixtures leading to the production of light building products, the surface Tension of the mixture is increased by addition of surface tensioning agent which does not chemically react with the mixture, thus allowing to trap the inert gas in small bubbles which are homogeneously distributed throughout the slurry mixture while the solidification takes place. After the consumption of water by the hydration process, voids and cavities are formed in the product structure. Aeration can be by mechanical mixing techniques or pulsating gas injection via a foaming dispenser. The foaming dispenser is a lance covered by a perforated rubber membrane that allows the gas, when under pressure only, to pass through the plurality of capillary holes.

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AERATED, LIGHTWEIGHT BUILDING PRODUCTS

Description

This invention relates to building products. More particularly although not exclusively it discloses an improved method of producing foamed masonry products using water based slurry mixtures.

The term "foamed" as used in this specification relates to the foaming up of these water based slurries by injection of an inert gas into the mixture under specific conditions of the ternary phase environment which will stabilise the cohesive and repulsive forces in such equilibrium to create a homogeneous dispersion system inseparable from its individual phase components.

One commonly used prior art method for producing foamed or aerated building products is to disperse aluminium powder (with or without cellulose derivatives) into the cementing mixture. The aluminium reacts with the alkali in the slurry to release hydrogen from aqueous solution. This expands the mixture while hardening takes place. Other chemical agents which are sometimes used are peroxides, sodium and potassium hydroxide. With such existing methods however the chemical agents used can be highly aggressive and dangerous to work with. Also the resulting chemical reactions can affect the equilibrium of the cementing reaction so that special post curing procedures must be used. These existing methods therefore tend to be

expensive and labour intensive. Quality control of the final product is also difficult.

It is therefore an object of this invention to ameliorate the aforementioned disadvantages and accordingly a method of producing a foamed masonry product is disclosed, said method including the steps of:-

- preparing a water based cement slurry mixture,
- introducing a predetermined amount of a surface tensioning agent into said mixture,
- introducing a volume of inert gas into said mixture in the form of minute bubbles sufficient to foam up and expand the volume of said mixture, and
- curing said mixture to produce a foamed product of predetermined density and specific gravity.

In another aspect this invention also discloses an injector for dispersing gas into a wet cement slurry mixture, said injector comprising one or more lance nozzles of perforated rubber membrane, said membrane being adapted to pass pressurised gas through a plurality of capillary holes into said mixture in the form of minute bubbles and upon cessation of gas flow said membrane being further adapted to close said holes to prevent backflow into said nozzles.

By injecting and trapping inert gas in a wet slurry mixture in accordance with this invention and keeping it there until the mixture hardens a product is created which is much lighter. The final volumetric gravity of the material is controlled by the volume of solids and liquid phase displaced by the gas.

Due to large differences in the specific gravities of the three phases the rapid separation of one from another by the action of gravity is inevitable, unless this force is counteracted by other means of attraction. This problem most affects the stabilisation of gas in ternary mixtures due to an approximate 1000 fold difference in the specific gravities of the gas and the other two phases. It can be partially overcome by controlling the parameters for the gas bubble generation environment described by the following Laplace equation

$$(p_{in} - P_{out}) = 2\ \gamma/r$$

This equation indicates that there are only two variables controlling the conditions under which gas can be restrained from separation in the slurry. These are the bubble size (r) and the Coefficient of Surface Tension (γ) of the fluid surrounding the bubble. The latter is the most critical function in the process.

The smaller the gas bubbles are, the greater the pressure differential between the inner and outer phase of bubbles and less gravitational buoyancy will affect the separation of phases. Also, the minute bubbles will support larger and heavier solid particles in the mixture, essentially acting like small ball bearings.

The higher the fluid surface tension is, the proportionally higher the pressure differential will be and the bubbles will tend not to agglomerate into larger sizes which have greater buoyancy effect. Therefore to achieve equilibrium between the gas and the other two phases in cementing slurry

mixtures the force which operates to contract the surface of the bubble must be increased by the addition of a surface tensioning agent (STA). The gas must also be injected in the form of the smallest possible bubble sizes. For process stability the injected gas and the STA must not chemically react with any of the slurry components. The STA must also be fully water soluble. In the majority of cases ambient air is a satisfactory inert gas for this application.

The following are three examples of production methods for manufacturing foamed building products according to this invention.

EXAMPLE 1 - The Two Part Process

This process is suitable for fast hardening cement slurry mixtures containing gypsum or curing accelerators where the process is time restrained. The process includes three steps.

(a)Preparation of fine foam which is produced separately with minimum amount of water mixed with the surface tensioning agent,

(b)Preparation of the cementing slurry mixture with an empirically determined amount of water, and

(c)Quickly mixing together ingredients (a) and (b) and then immediately casting the mixture into moulds.

EXAMPLE 2 - Mechanical Foaming Process

This method is suitable for preparation of foamed mixtures containing small solid particles such as sand, clay or ground rock with cementing binders.

The cementing slurry mixture is dosed with an empirically determined amount of a surface tensioning agent. Ambient air is then introduced into the mixture by a high speed mechanical agitator which may be similar to a commercial cake mixer. The specific gravity of the product is controlled by measuring the increase in volume of the known weight of the mixture or by sampling for specific gravity during the mixing process.

One disadvantage of mechanical mixing methods is the rapid wear of interfacing parts of the mixer due to the abrasive action of the solid materials at the very high mixing speeds required.

<u>EXAMPLE</u> 3 - The Gas Injection Process

This method is most suitable for cementing slurry mixtures with larger sized aggregates such as concrete. However, it will also be satisfactory for use in all three outlined methods. Inert gas is injected into mildly agitated (slowly mixed) cementing slurry via a stationary lance with a foaming dispenser. Mixing of the slurry during the gas injection operation is required to achieve a even distribution of suspension in the mixture.

More specifically, after addition of the STA into the slurry the inert gas (e.g. air) is pulsed through a large number of small capillary holes with a fully submersed portion of the foaming dispenser. This arrangement generates large numbers of individual small bubbles which distribute evenly through the slurry. The injected bubbles are subjected to a sh aring action by the mixing flow which tends to further cut the bubble sizes into smaller volumes. The larger the

currently preferred design of the gas dispenser is shown in figure 1. Specific gravity of the product is controlled by sampling the slurry, or by measuring the amount of gas (air) introduced into it. If there are no gas losses during the injection process then all gas will be trapped in the slurry and the measuring method becomes simple and reliable. Should the mixing vessel enable accurate measurements of the slurry volume then this method is also applicable.

Examples of the above methods are outlined in more detail in the appendix.

DESCRIPTION OF THE FOAMING SYSTEM

Referring to figure 1, compressed gas 1 is charged into a reducing valve 2. The gas at reduced pressure is fed into a solenoid controlled valve 3 which oscillates (e.g. at say 50 Hz). Electrical safety on site is preferably achieved by the power supply voltage being reduced by the transformer 4. The injected gas flow is measured by a flow meter 5. A pressure gauge 6 measures the hydrostatic head acting upon the gas at discharge. From the control system the gas is fed to a lance 7 by a connecting hose 8. The lance 7 is held in a central position by a support device 8 bolted to a shute. The foaming dispenser 9 must be submersed under the slurry mixture 10 to avoid gas discharge into the atmosphere. The truck 11 may have to reverse up a ramp 12 if the volume of the slurry is low and the foaming dispenser 9 would not be otherwise covered.

The foaming dispenser is preferably made of perforated strong soft rubber which disperses the gas freely when the

pressure difference between the supply side is higher then the outlet side. Due to the resiliency of the rubber the capillary holes of the dispensing device automatically close when the supply pressure drops below the hydrostatic pressure of the slurry. The dispenser as well as the gas supply system is thus protected against accidental blockage by the fine particles of the cementing mixture. This provides a clear advantage over capillary dispensers with permanently open cells.

The dispensing volume per single perforation in the dispenser maybe calculated for bubble sizes of maximum diameter 1mm which corresponds to a volume of 0.5236 mm^3 At the oscillating valve speed frequency of preferably 50 Hz the gas volume discharged through one hole will be a maximum of 1.57 cm^3 /min. Therefore to obtain a gas discharge of 100 l/min at this rate 63694 perforations will be required. This represents at 100 holes/cm^2 a dispensing area of 636.94 cm^2 (6.37 dm^2) . For correct gas volume injection in relation to the final specific gravity of the product compensation must be made for the slurry temperature and average hydrostatic pressure.

Referring now to figure 2 the feeder lance 7 is connected to the lance nozzle shell 13 by a joiner 14. The gas is discharged through a perforated rubber membrane 15. The number of holes in the rubber membrane determines the number of bubbles which will be discharged into the slurry mixture. The rubber membrane 15 is held in place by the support 16. A gas-tight joint to the rubber membrane is obtained by a compression seal between the joiner 14 and support 16. The gas discharge capacity can be increased by multiple foaming dispensers joined together in series

provided that each stays submersed in the slurry. The end of the foaming dispenser is protected by a nose cone 17 which abuts the rotating centre of the mixer.

The concentration of the STA in cementing slurries will mainly vary depending upon the cementing reaction speed and the amount of unreacted water available to form the skin of the bubbles. Inversely, the amount of excess water in the slurry is controlled by the stochiometric amount required for the cementing (hydration) reaction and the surface adsorption characteristics of all solid ingredients in the mixture (wetting or soaking). With higher concentrations of finer solid particles a greater amount of water is required to foam up the mixture. Therefore for each STA and each mixture having a different composition of ingredients laboratory tests will guide the selection of the optimum dose. Obviously those STA's with higher surface tensions will be added in proportionally smaller amounts than those with lower surface tension characteristics. However, tests with water soluble STA's indicate that the maximum required concentrations of STA's with fairly low surface tensions just above 0.1 N/m are in the order of 1% of the total weight of the cementing mixture. Some of the STA's with higher surface tensions will do the same job at concentrations less than 0.1%. Therefore in practical terms values between lkg to 10 kg of STA's per 1000 kg of the product enable a safe expansion in some cases up to 4.6 times the original volume.

Foamed building products will not support the same compressive load as solid building materials as the compressive strength is weakened by cavities within the

mass. For this reason their application should be for limited structural or non structural use.

During the process of solidification, the water in the mixture is gradually used up for the hydration reaction. This opens the voids in the product where the bubbles originally occupied space among the solid particles. It results in an open cell structural formation with the larger solid particle distribution contained in the original mixtures. These foamed building products satisfy the physical requirements for sound absorption materials with the internal resonance characteristics introduced by the open cell structural webs. As the acoustic absorption characteristics of these products can be precisely controlled, they may be used in the design of sound barriers, auditory chambers, theatrical and conference rooms, walls in high rise buildings and other applications where reduction of sound transmission or sound reverberation is important.

Foamed building products produced from slurry mixtures containing higher concentrations of pulverised solids (below 0.1 mm) will maintain the spherical shapes of bubble cavities in the final product during the solidification process. The drying up time of the unreacted excess water content in such mixture will be prolonged. These foamed building products harden with closed cellular structures without acoustic absorption properties. However, they are sealed for prolonged resistance against air and water entry into the material and have improved compression strengths in comparison to open cell structure materials. These products are more suitable for external building applications. They do not require surface rendering and can

be used as self supporting structural walls for lower compression strength applications.

APPENDIX

The aim is to obtain a product of the Final Specific Gravity (FSG) with easy control of this parameter. Due to the fact that the mixture compositions can vary considerably depending on applications, for each case the amount of gas injection will also be different in order to achieve the same result. The decisive controlling factor for the required gas volume is the injected gas temperature with the combined liquid+pipe backpressure and the temperature of the aqueous mixture at average hydrostatic pressure of the slurry where the gas is injected to. The calculation of the required gas volume at injection gas conditions are expressed in modified equation of State:

$$GV = \frac{P2(1+0..003663\,t1) \times \left\{ \dfrac{\Sigma w - [FSG(\Sigma v)]}{FSG} \right\}}{P1(1+0.003663t2)}$$

Numerical inputs into the Example calculation:

P1 = 1000 mm Hg
P2 = 900 mm Hg
FSG= 1.2
Σw = 4.66 tons
Σv = 2.36 m^3
t1 = 20 deg.C
t2 = 32 deg.C

Where:

GV = Required gas volume at injection point conditions
P1 = Gas pressure at gas injection measurement point
P2 = The average hydrostatic pressure of slurry
FSG= The Final Specific Gravity of the LBP to be achieved
Σw = The total weight of all materials in the slurry such as
[Sw(solids unreactive)+ Cw(cementing reactive)+ H_2O]
Σv = The total volume of all materials in the uninflated slurry as

[(Sw/SG + Sw/SG + H_2O)]

t1 = The gas temperature at the injection point, deg. C

t2 = The slurry temperature, deg. C.

The numerical Example using the above figures.

$$GV = \frac{900(1+0.003663\text{x}20)\ \text{x}\ \left\{ \dfrac{4.66 - (1.2\text{x}2.36)}{1.2} \right\}}{1000(1+0.003663\text{x}32)} = 1.317\ m^3 \text{ of gas}$$

measured at injection point

DESCRIPTION OF METHOD 1

With reference to figure 3, the foam is prepared by minimal required volume of aqueous solution with STA in the mixer 18 using the high speed foam starter 19. The continuation of the foaming process is done by a variable speed foaming peddle system 20. The volume of foam is controlled by the level detector 21. Once the desired level of foam is obtained the cementing slurry is prepared in the bin 22. The dose is weighted with a scale 23 and the slurry is mixed with the foam to form a homogenous mix which is then discharged by valve 24. The vessel is then ready for another batch.

DESCRIPTION OF METHOD 2

With reference to figure 4, the dry solids and cements are mixed in bin 25 and weighted with scales 26 then discharged into mixer 27 together with the measured water and STA from tank 28 via hose 29. The mixing process starts with the slurry level at position 30 with the slow rotations of the mixing paddles 31 and gradually with the reduction of SG

the mixing speed is raised. The mixing is stopped when the expanded volume reaches the required level measured with sensor 32. Then the finished mixture at final SG is discharged through valve 33 with a slow agitation speed of the paddles 31. The system is driven by a variable speed motor 34 through a reduction gearbox 35.

Claims

The claims defining the invention are as follows:

1. A method of producing a foamed masonry product, said method including the steps of:-

- preparing a water based cement mixture,
- introducing a predetermined amount of a surface tensioning agent into said mixture,
- introducing a volume of an inert gas into said mixture, in the form of minute bubbles sufficient to foam up and expand the volume of said mixture, and
- curing said mixture to produce a foamed product of predetermined density and specific gravity.

2. The method as claimed in claim 1 wherein said inert gas is introduced in the form of a fine foam comprising water and said surface tensioning agent.

3. The method as claimed in claim 1 wherein said cement slurry mixture is dosed with said predetermined amount of surface tensioning agent and said inert gas is subsequently introduced by high speed mechanical agitation.

4. The method as claimed in claim 1 wherein said cement slurry mixture is dosed with a predetermined amount of said surface tensioning agent and said inert gas is subsequently introduced by means of a foaming dispenser while the mixture is mildly agitated.

5. The method as claimed in claim 4 wherein said inert gas is pulsed through a large number of small capillary holes in said foaming dispenser.

6. An injector for dispersing inert gas into a wet cement slurry mixture, said injector comprising one or more lance nozzles of perforated rubber membrane, said membrane being adapted to pass said inert gas under pressure through a plurality of capillary holes into said mixture in the form of minute bubbles and upon cessation of gas flow said membrane being further adapted to close said holes to prevent back flow into said nozzles.

7. The injector as claimed in claim 6 wherein said capillary holes are sized to disperse bubbles of no more than 1 mm in diameter.

8. The injector as claimed in claim 6 or 7 wherein there are two or more nozzles connected end to end in series.

9. A method of producing a foamed masonry product, said method being substantially as described herein with reference to the examples.

10. An injector for dispensing inert gas into a wet cement slurry mixture, said injector being substantially as described herein with reference to figures 1 and 2.

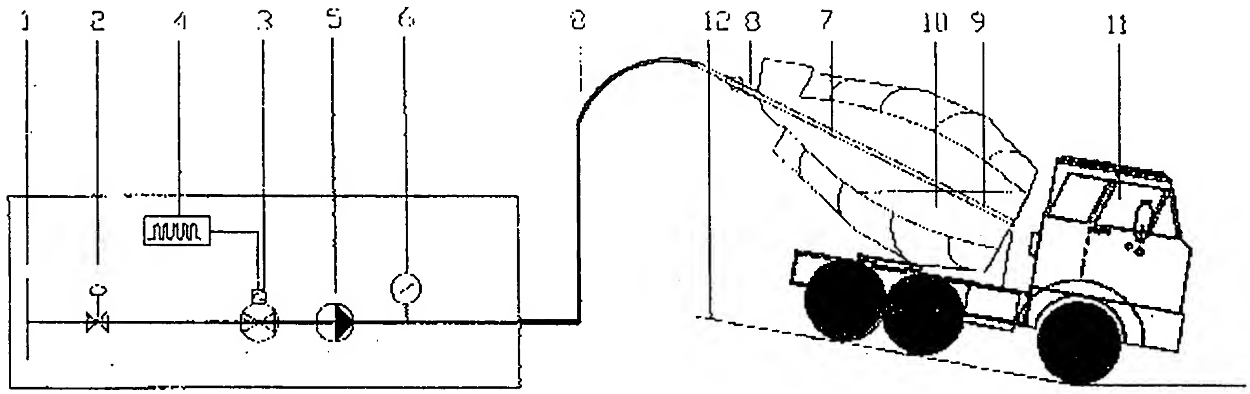

Fig. 1

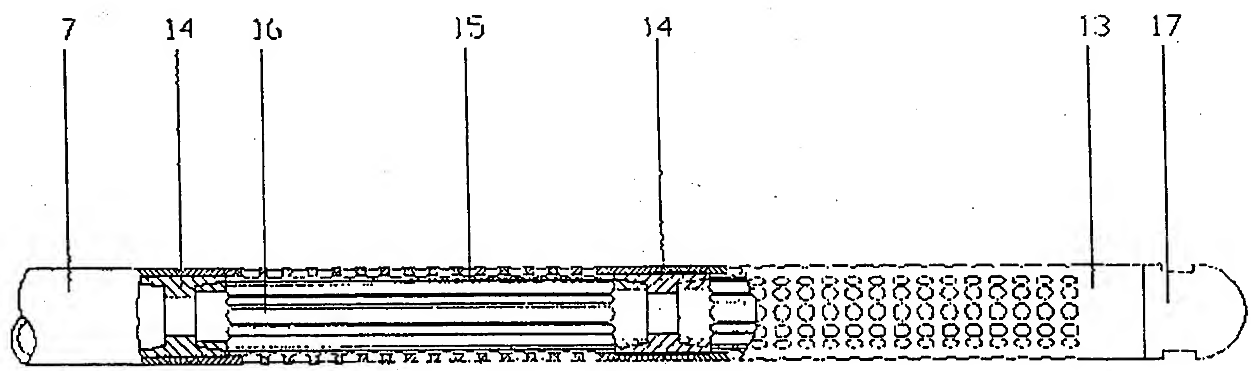

Fig. 2

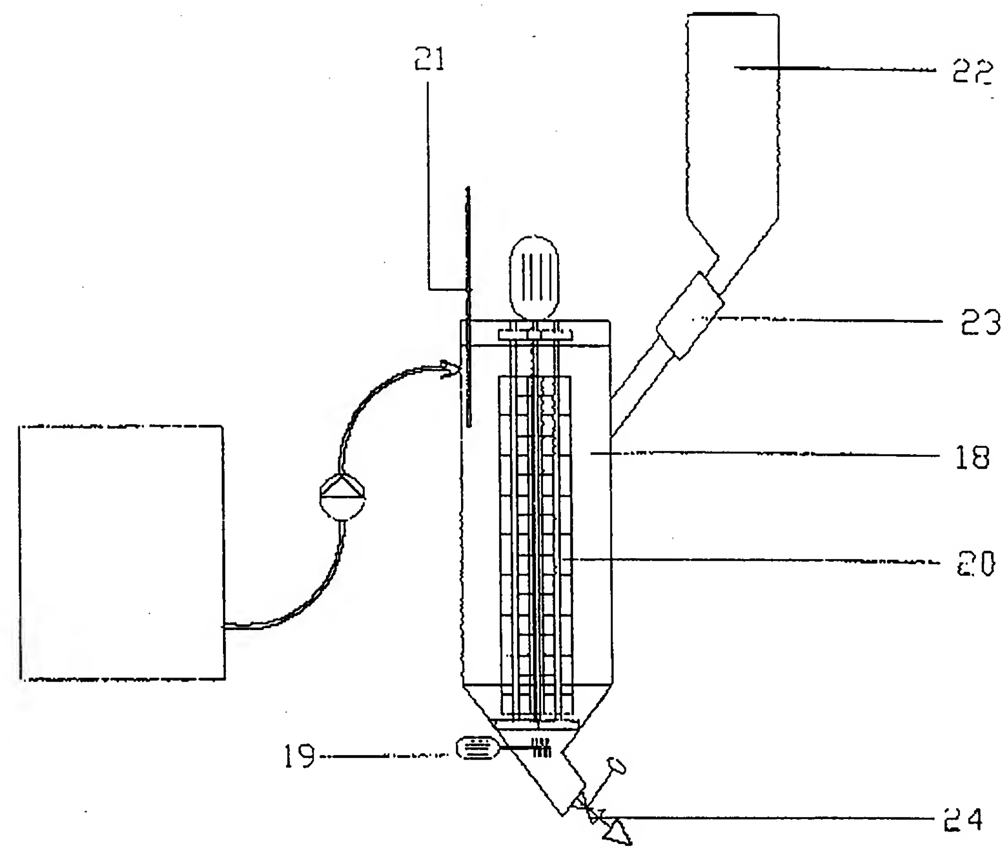

Fig. 3

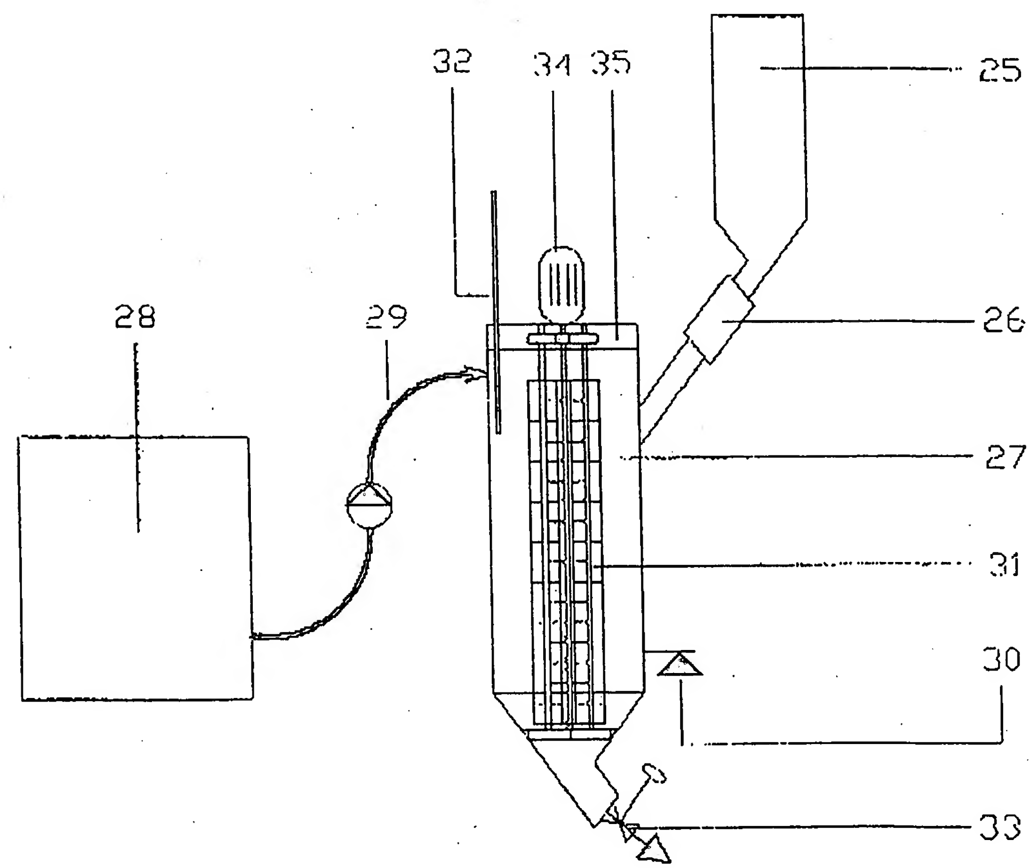

Fig. 4

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 98/00196

A. CLASSIFICATION OF SUBJECT MATTER

Int Cl[6]: C04B 38/00; B01F 3/04, 5/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC[6]: C04B 38/00; B01F 3/04, 5/04
IPC[1-3]: C04B 21/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
AU: IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4415366 A (COPENING), 15 November 1983	1
X	US 5588489 A (CHATTERJI et al), 31 December 1996	1
X	US 4789244 A (DUNTON et al), 6 December 1988	1
X	GB 2086748 A (DOMOCON SA), 19 May 1982	1,3
X	FR 2503617 A (DION-BIRO), 15 October 1982	1
X	US 3989534 A (PLUNGUIAN et al), 2 November 1976	1

[X] Further documents are listed in the continuation of Box C

[X] See patent family annex

* Special categories of cited documents:

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"E" earlier document but published on or after the international filing date

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
8 July 1998	13 JUL 1998

Name and mailing address of the ISA/AU	Authorized officer
AUSTRALIAN PATENT OFFICE PO BOX 200 WODEN ACT 2606 AUSTRALIA Facsimile No.: (02) 6285 3929	DAVID LEE Telephone No.: (02) 6283 2107

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 98/00196

Box I **Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)**

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box II **Observations where unity of invention is lacking (Continuation of item 2 of first sheet)**

This International Searching Authority found multiple inventions in this international application, as follows:

See attached

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest ☐ The additional search fees were accompanied by the applicant's protest.

☒ No protest accompanied the payment of additional search fees.

Box II (continued)

1 Claims 1-5 refer to a method of making a foamed masonry product by performing several steps on the cement mixture. The method does not require an injector (like that of claim 6) to introduce inert gas into the mixture. Claims 1-5 result in a "foamed" masonry product.

It is known to introduce inertgas (eg nitrogen or air) into a cement slurry, with a surfactant, to produce "foamed" masonry products. (See the following patent specifications as examples only, US 5588489 (31 December 1996), US 4300633 (17 November 1981), DE (German) 4041727 (2 July 1992), JP 04/255303 (10 September 1992), DE 4126397 (11 February 1993) and many more).

2 Claims 6-10 refer to an injector (suitable for injecting gas into a wet cement slurry). An injector is not the same as making a foamed masonry product.

3 These two sets of claims do not share novel, common subject matter. As the subject matter of claim 1 is already anticipated, the two sets of claims do not share a single inventive concept.

4 Consequently, there is lack of unity of invention (more than one invention) defined by the two sets of claims. These claims do not fulfil PCT rule 13.2.

C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 3319161 A1 (MENZEL GmbH), 29 November 1984	6
X	DE 3418548 A1 (SCHNEIDER), 21 November 1985	6
X	US 5059358 A (THARP), 22 October 1991	6
X,Y	WO 92/00798 A1 (LANMARK CONSULTANTS), 23 January 1992	6,7
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X,Y	DE 2940407 A1 (BUCHHOLZ), 9 April 1981	6,7
X	US 4489016 A1 (KRIEBEL), 18 December 1984	6

INTERNATIONAL SEARCH REPORT
Information on patent family members

International Application No.
PCT/AU 98/00196

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
US	4415366	NONE					
US	5588489	CA	2189215	EP	771768	NO	964583
		US	5711801				
US	4789244	US	2872913				
GB	2086748	CA	1162949	US	4383862		
FR	2503617	NONE					
US	3989534	NONE					
DE	3319161	NONE					
DE	3418548	NONE					
US	5059358	CA	2042653				
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FR	2750889	NO	962328	NO	972520		
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		IT	1132913	JP	56058527	NL	8005495
US	4489016	NONE					

END OF ANNEX